Fracture And Deformation Damage Accumulation In Tough Ceramics

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Abstract

A study on the role of microstructure in the contact damage and fatigue of tough ceramics, using *in-situ*-toughened silicon nitride as a case study, is described. The thrust of the study is on fundamental mechanics of damage accumulation in relation to microstructural design. A focal point is the characterization and evaluation of new quasi-plastic modes of damage, in addition to the conventional fracture modes, that can limit the long-term performance of tougher ceramics under concentrated loading conditions. Ultimate goals are as follows: (i) to investigate the interrelations between these damage modes and associated mechanical properties, notably strength properties and fatigue resistance; (ii) to develop new silicon nitrides with controlled microstructures for high resistance to damage accumulation; (iii) to fabricate layer structures for maximum toughness *and* wear resistance. Implications concerning the design of tough materials for bearing applications and coating-substrate technologies will be discussed.

I. Introduction

It is well recognized that the toughness of silicon nitride and other ceramics can be profoundly increased by suitably tailoring the microstructures, e.g. by growing large elongated grains [1-4] and by manipulating the grain boundary phase [5,6], in order to enhance bridging [4,7-9]. However, any such toughness increase is generally limited to the "long-crack" region. Properties determined at the microstructural level, among them strength and fatigue resistance, may undergo simultaneous degradation [10-13]—in extreme cases such degradation may be sufficiently pronounced as to render the ceramic "machinable" [14]. At issue here is how increased heterogeneity in the microstructure affects damage accumulation in the "short-crack" region. There is a clear need to understand the role of internal microstructural variables in determining the nature and severity of such damage accumulation, and how this accumulation controls lifetime characteristics.

Nowhere is the microstructural interaction between materials variables and damage properties more important than in contact fields, where ceramic surfaces may be subjected to intense local stress concentrations over long periods of time, and over millions of loading cycles. Such concentrated stresses are especially pertinent to bearing applications. Ceramic engine components are also susceptible to concentrated loads. Recent studies using spherical indenters have provided new insights into the nature of contact damage [15-19], especially in cyclic loading [20,21]. These studies reveal that the mode of the damage can change fundamentally with modifications to the material microstructure, from a classical tensile-driven single cone fracture ("brittle" response) in fine-grain homogeneous ceramics to distributed shear-driven micro-deformation in coarse-grain heterogeneous ceramics ("quasi-plasticity") [22,23]. The brittle and quasi-plastic responses have different influences on strength loss and material removal properties of the material, but both are ultimately deleterious. Because it occurs subsurface, the quasi-plastic mode has hitherto passed unnoticed in bearing technology. However, it can have a profoundly deleterious effect on effective lifetimes of components in bearing and other applications, especially under fatigue conditions. This raises the question: what are the optimal microstructural properties for minimizing susceptibilities to brittle and quasi-plastic damage?

In this paper we describe a study of Hertzian contact on tough ceramics, and show how increasing microstructural heterogeneity can lead to an effective brittle-to-ductile transition. We demonstrate the role of microstructure in a case study on silicon nitrides with ever-coarsening and elongating microstructures, although we emphasize the generality of the method [23]. We demonstrate how tougher silicon nitrides, while damage tolerant in single-cycles, are especially susceptible to fatigue in multiple cycles. Indications as to the construction of micromechanical models for quantifying the damage are

given. Finally, we describe how combinations of material types may be designed in layer structures to provide maximum damage and wear resistance.

II. Contact Damage and Fatigue in Hertzian Contact

We consider three silicon nitrides, all from the same initial composition but heat treated at different times and temperatures [24,25]: (a) a fine (F) structure with predominantly equiaxed α grains (1570°C for 1 h); (b) an intermediate (M) structure with some equiaxed α grains plus some in-situ grown elongated β grains (1650°C for 1 h); (c) a coarse (C) structure with predominantly elongated β grains (1800°C for 3 h). Indentations are made with WC spheres, radius 1.98 mm. Figure 1 compares the contact damage in these three microstructures. Whereas the cone crack pattern in the F material has all the hallmarks of a traditionally brittle response, the damage pattern in the C material resembles more the macroscopic features of a plastic zone in a ductile metal. The latter quasi-plastic damage initiates subsurface, and is not easily detected by routine surface inspection at the lower loads—here it is revealed by a "bonded-interface" pre-sectioning technique [17] (Sect. 3.1). Interestingly, bearing grade materials tend closest to the M structure shown here, where the fracture and deformation modes are in some degree of balance.

That both damage modes can be highly deleterious to performance of ceramics, especially under fatigue conditions, and particularly in water, has been clearly demonstrated by measuring the remaining strengths of bar specimens after contact damage [16-18,20,21,26]. Data illustrating how the strengths of the silicon nitrides in Fig. 1 degrade after a critical number of contact cycles are plotted in Fig. 2, for contacts at loads (a) well below and (b) well above that required to produce any detectable damage in a single cycle. The ultimate strength losses are particularly severe in the F structure, from abrupt popin of dominant cone crack flaws. However, they are also substantial in the C structure, if less abrupt, from microdamage flaws within the quasi-plastic zone, especially at high loads and large numbers of cycles, as the microdamage flaws extend and coalesce with neighbors [21] (see below). Note that the M structure shows relatively moderate degradation at large n, indicating an offsetting compromise between the fracture and quasi-plasticity damage modes. Again, it is of interest that this M structure most closely simulates that used in bearing grade silicon nitrides.

Work on elucidating the role of other microstructural variables in the silicon nitride system by controlling the $\alpha-\beta$ phase ratio (e.g. by adjusting the starting powders), grain boundary phase, etc., is in progress [27].

Mathematical models for describing Hertzian contact damage in tough ceramics like silicon nitrides are currently being developed [25,28-32]. The form of damage in homogeneous brittle ceramics is the classical cone fracture. This crack initiates in a zone of weak tensile stress *outside* the contact circle, and flares downward and outward into the material as a well-defined cone. In heterogeneous ceramics with coarse grains and weak interfaces the incipient cone cracks are suppressed, by deflecting along the weak interfaces away from the tensile stress directions, and are unable to propagate. Instead, as a result of the high shear stresses *below* the contact area, discrete grain-localized deformation is activated at weak internal interfaces (interphase boundary sliding, intragrain twinning), allowing the material to "yield". These so-called "shear faults" concentrate stresses at their ends, and in turn generate subfacet microcracks ("wing" cracks) along the weak boundaries. The collective influence of these faults and microcracks are characterized by a damage parameter Nc^3 ($N = Nc^3$) number density of damage events, $C = Nc^3$ mean scale of event) [32]. The fault–microcrack flaws degrade the material in the subsurface region, and lead to strength loss [31].

These models are only now being extended to cyclic loading conditions. In brittle ceramics, where the fatigue effect is essentially chemical, analysis simply requires the incorporation of an appropriate crack velocity relation v = v(K) into the cone fracture mechanics [21]. In quasi-plastic ceramics mechanical processes dominate—damage accumulates primarily by decremental attrition at the sliding shear fault interfaces, manifested as a progressive reduction in frictional sliding resistance [28,29]. Complex interactions between chemical and mechanical effects can occur (e.g. by reducing the friction at shear faults and enhancing the extension of attendant microcracks), although these micro-interactions are not yet well understood.

III. Silicon Nitride Layer Structures

Work has also begun on developing silicon nitride and other ceramic layer structures, with the aim of producing laminar composites with hard, wear-resistant outer surfaces and soft, tough underlayers [33-35]. A unique feature of such layer composites is the incorporation of strong interlayer interfaces, to avoid delamination. Initial work has demonstrated that layered structures of this kind, as well as providing surface hardness, can greatly suppress subsurface fractures. Examples of contact damage produced by a WC indenter in such a structure are shown in Fig. 3, with a common homogeneous top layer ("coating") on two heterogeneous underlayers with quite different toughness properties ("substrates") [35]. In its bulk state (Fig. 3a) the coating is a relatively fine Si₃N₄ (cf. Fig. 1a) with well-developed cone fracture. The substrate in the first bilayer (Fig. 3b) is a coarse but pure Si₃N₄ (cf. Fig. 1c), corresponding to modest coating/substrate elastic-plastic mismatch. A cone fracture is still evident, but is significantly shallower. Note the appearance of a deformation zone beneath the indenter. The substrate in the second bilayer (Fig. 19c) has a much more heterogeneous microstructure, with incorporated boron nitride platelets as a softening phase [34], corresponding to a large coating/substrate

elastic—plastic mismatch. The damage pattern is much more complex, with multiple transverse coating fractures extending both downward from the top surface and upward from the interlayer interface. Substrate yield is now pronounced. It is noteworthy that the transverse cracks again remain fully contained within the coating in both bilayers—extreme loads are needed to drive these cracks through the coating thickness and cause failure (attesting to the damage tolerance of these structures)—and that there is no substantial delamination at the interlayer interface. It is clear that the mismatch has a profound influence on the coating fracture.

IV. Conclusions

We have demonstrated the practicality of contact with spheres in the characterization of damage modes in ceramics under concentrated loading conditions, using silicon nitride as a case study. In the tougher ceramics, quasi-plasticity supplants cone fracture as the dominant mode of damage. Tougher ceramics are also subject to accelerated fatigue in repeat loading, from accelerated damage accumulation. Combining hard–brittle and soft-tough ceramics into coating/substrate layer structures with strong interlayer interfaces offers the prospect of enhancing toughness without sacrificing wear resistance.

References

- 1. F.F. Lange, J. Am. Ceram. Soc. 62, 428 (1979).
- 2. C.-W. Li and J. Yamanis, Ceramic Engineering and Science Proceedings 10, 632 (1989).
- 3. C.-W. Li, D.-J. Lee and S.-C. Lui, J. Am. Ceram. Soc. 75, 1777 (1992).
- 4. C.-W. Li, S.-C. Lui and J. Goldacker, J. Am. Ceram. Soc. 78, 449 (1995).
- 5. P.F. Becher, S.L. Hwang, H.T. Lin and T.N. Tiegs, *Tailoring of Mechanical Properties of Si₃N₄*, (edited by M.J. Hoffmann and G. Petzow), p. 87. Kluwer Academic Publishers, Dordrecht, The Netherlands, (1994).
- 6. M.J. Hoffmann, *Tailoring of Mechanical Properties of* Si_3N_4 , (edited by M.J. Hoffmann and G. Petzow), p. 59. Kluwer Academic Publishers, Dordrecht, The Netherlands, (1994).
- 7. P.L. Swanson, C.J. Fairbanks, B.R. Lawn, Y.-W. Mai and B.J. Hockey, J. Am. Ceram. Soc. 70, 279 (1987).
- 8. S. Lathabai, J. Rödel and B.R. Lawn, J. Am. Ceram. Soc. 74, 1340 (1991).
- 9. B.R. Lawn, Fracture of Brittle Solids, Second, Cambridge University Press, Cambridge (1993).
- 10. S.J. Bennison and B.R. Lawn, Acta Metall. 37, 2659 (1989).
- 11. S.J. Bennison, J. Rödel, S. Lathabai, P. Chantikul and B.R. Lawn, *Toughening Mechanisms in Quasi-Brittle Materials*, (edited by S.P. Shah), p. 209. Kluwer Academic Publishers, Dordrecht, The Netherlands, (1991).
- 12. B.R. Lawn, N.P. Padture, L.M. Braun and S.J. Bennison, J. Am. Ceram. Soc. 76, 2235 (1993).
- 13. N.P. Padture, J.L. Runyan, S.J. Bennison, L.M. Braun and B.R. Lawn, J. Am. Ceram. Soc. 76, 2241 (1993).
- 14. N.P. Padture, C.J. Evans, H.H.K. Xu and B.R. Lawn, J. Am. Ceram. Soc. 78, 215 (1995).
- 15. F. Guiberteau, N.P. Padture, H. Cai and B.R. Lawn, Philos. Mag. A 68, 1003 (1993).
- 16. H. Cai, M.A. StevensKalceff and B.R. Lawn, J. Mater. Res. 9, 762 (1994).
- 17. F. Guiberteau, N.P. Padture and B.R. Lawn, J. Am. Ceram. Soc. 77, 1825 (1994).
- 18. H.H.K. Xu, L. Wei, N.P. Padture, B.R. Lawn and R.L. Yeckley, J. Mater. Sci. 30, 869 (1995).
- 19. N.P. Padture and B.R. Lawn, J. Am. Ceram. Soc. 77, 2518 (1994).
- 20. H. Cai, M.A.S. Kalceff, B.M. Hooks, B.R. Lawn and K. Chyung, J. Mater. Res. 9, 2654 (1994).
- 21. N.P. Padture and B.R. Lawn, J. Am. Ceram. Soc. 78, 1431 (1995).
- 22. B.R. Lawn, N.P. Padture, H. Cai and F. Guiberteau, Science 263, 1114 (1994).
- 23. B.R. Lawn, J. Am. Ceram. Soc. (in press).
- 24. S.K. Lee, S. Wuttiphan and B.R. Lawn, J. Am. Ceram. Soc. 80, 2367 (1997).
- 25. S.K. Lee and B.R. Lawn, J. Am. Ceram. Soc. (in press).
- 26. A. Pajares, L. Wei, B.R. Lawn and D.B. Marshall, J. Mater. Res. 10, 2613 (1995).
- 27. S.K. Lee, K.S. Lee, B.R. Lawn and D.K. Kim, J. Am. Ceram. Soc. (in press).
- 28. B.R. Lawn, N.P. Padture, F. Guiberteau and H. Cai, Acta Metall. 42, 1683 (1994).
- 29. N.P. Padture and B.R. Lawn, Acta Metall. 43, 1609 (1995).
- 30. A.C. Fischer-Cripps and B.R. Lawn, J. Am. Ceram. Soc. 79, 2609 (1996).
- 31. B.R. Lawn, S.K. Lee, I.M. Peterson and S. Wuttiphan, J. Am. Ceram. Soc. (in press).
- 32. B.R. Lawn and D.B. Marshall, J. Mech. Phys. Solids 46, 85 (1998).
- 33. L. An, H.M. Chan, N.P. Padture and B.R. Lawn, J. Mater. Res. 11, 204 (1996).
- 34. K.S. Lee, S. Wuttiphan, X.Z. Hu, S.K. Lee and B.R. Lawn, J. Am. Ceram. Soc. (in press).
- 35. K.S. Lee, S.K. Lee, B.R. Lawn and D.K. Kim, J. Am. Ceram. Soc. (in press).

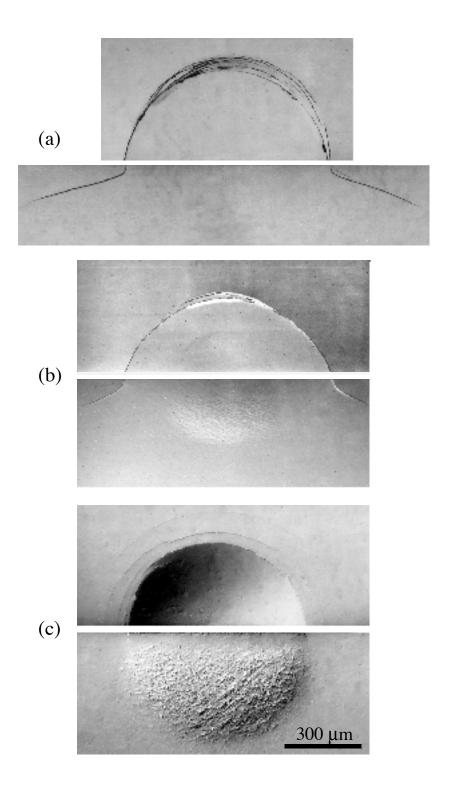


Fig. 1 Half-surface and side views of Hertzian contact damage in (a) F-Si₃N₄, (b) M-Si₃N₄, (c) C-Si₃N₄, from WC sphere radius r = 1.98 mm at load P = 4000 N. Nomarski optical micrographs of bonded-interface specimens. Note transition from fracture-dominated to quasi-plasticity-dominated damage pattern through F-M-C.

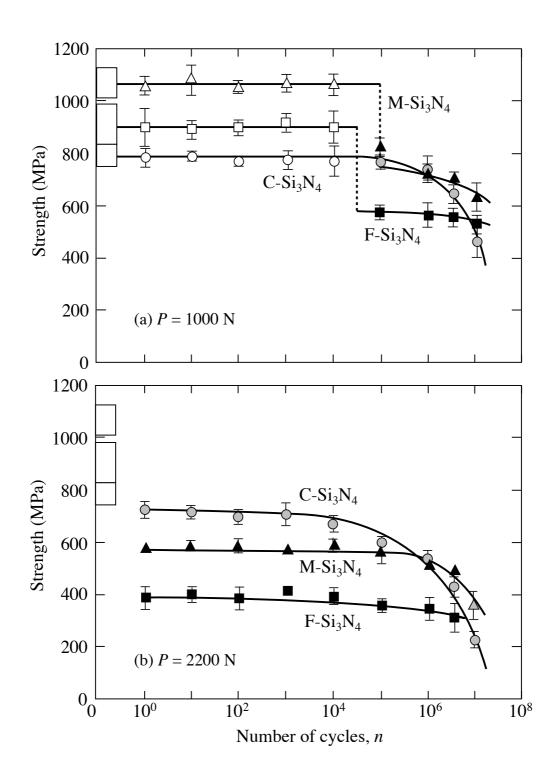


Fig. 2 Strength as a function of number of contact cycles for $F-Si_3N_4$, $M-Si_3N_4$, $C-Si_3N_4$, demonstrating role of microstructure. Indentation with WC spheres, r=1.98 mm, in air: at loads $P=(a)\ 1000$ N and (b) 2000 N. Black symbols indicate failure from cone cracks, grey symbols from quasi-plastic zones, and open symbols from natural flaws. Boxes at left axis indicate "laboratory" strengths.

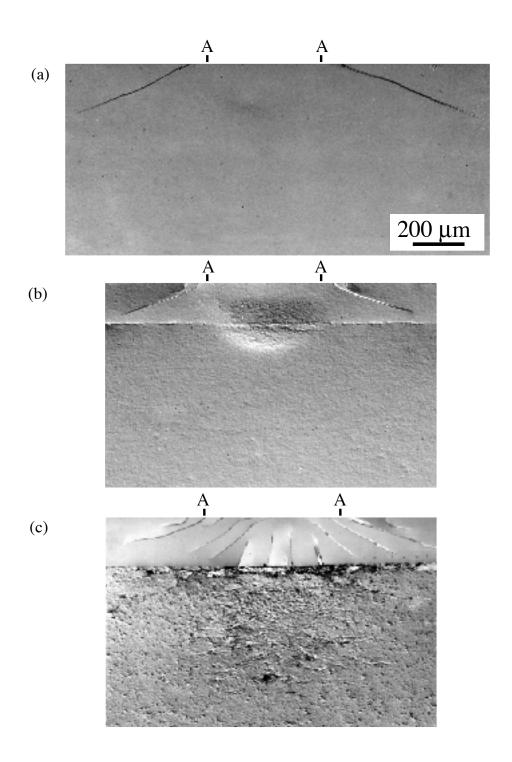


Fig. 3 Section views of Hertzian contact damage in $\mathrm{Si}_3\mathrm{N}_4$ bilayers, using WC sphere (r=1.98 mm, P=3000 N): (a) fine-grain monolith of fine-grain material, showing fully developed cone crack with barely detectable subsurface quasi-plasticity; (b) bilayer of fine-grain coating on coarse-grain substrate; (c) similar, but on substrate with 30% boron nitride additive. Bonded-interface specimens, Nomarski optical micrographs. Contact diameter AA indicated.